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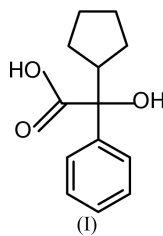
## Key indicators

Single-crystal X-ray study  
 $T = 142\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.036  
 $wR$  factor = 0.099  
Data-to-parameter ratio = 13.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.2-Cyclopentyl-2-hydroxy-2-phenylacetic acid,  
a pseudosymmetric crystal structure with  
space group  $Pna2_1$  and  $Z' = 2$ 

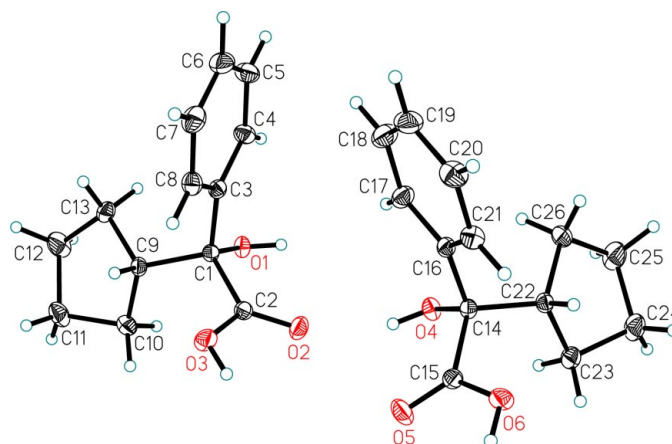
The title compound,  $\text{C}_{13}\text{H}_{16}\text{O}_3$ , crystallizes in space group  $Pna2_1$  with  $Z = 8$ . The two independent molecules are related by pseudosymmetry elements. The structure can be derived from the higher symmetric space group  $Pnam$  by translating the  $x$  coordinates of molecule 2 by  $-1/4$  with respect to those of molecule 1.

## Comment

The title compound, (I), was prepared as a starting material for the synthesis of possible anticholinergic agents.

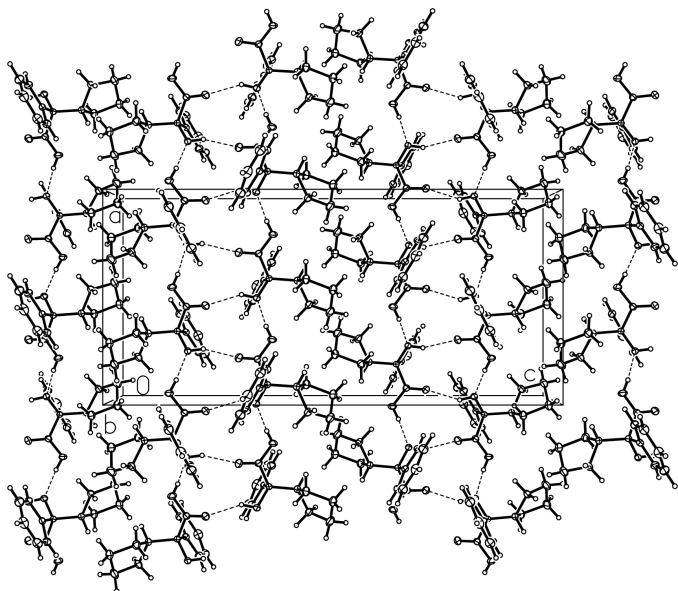


The structure contains two crystallographically independent molecules (Fig. 1). The dimensions of both molecules are very similar. The maximum differences for corresponding bond distances and angles are  $0.021\text{ \AA}$  for the  $\text{C}2-\text{O}2$  and  $\text{C}15-\text{O}5$  bonds, and  $0.9^\circ$  for the  $\text{C}6-\text{C}7-\text{C}8$  and  $\text{C}19-\text{C}20-\text{C}21$  angles. Corresponding torsion angles in the two molecules agree within  $2^\circ$ . The phenyl groups are planar. The cyclopentyl rings have approximately envelope conformations: a  $\text{C}10$ -envelope for molecule 1 and a  $\text{C}23$ -envelope for molecule 2. The molecular conformation of (I) is very similar to the

**Figure 1**

The structures of the two independent molecules of (I), shown with 50% probability displacement ellipsoids.

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**Figure 2**  
The crystal packing of (I), viewed down *b*.

conformation of a 1,1-dimethylpyrrolidinium ester of (I), reported by Guy & Hamor (1973). Only the conformation of the cyclopentyl group is considerably different in that compound.

The molecules are connected by  $O_{\text{carboxyl}}-\text{H}\cdots O_{\text{hydroxyl}}$  hydrogen bonds to form chains in the crystallographic *a* direction (Table 1 and Fig. 2). One chain consists only of molecule 1, while the next chain consists only of molecule 2. The molecules within each chain are related by crystallographic *a*-glide-plane symmetry. Two neighboring chains are linked by  $O_{\text{hydroxyl}}-\text{H}\cdots O_{\text{keto}}$  hydrogen bonds to form double chains. These double chains are further stabilized by weak intermolecular  $\text{C}-\text{H}\cdots\pi$  interactions between phenyl groups:  $\text{C5}-\text{H5}\cdots\text{Cg}_2$  (where  $\text{Cg}_2$  is the centroid of the phenyl ring of molecule 2) and  $\text{C18}-\text{H18}\cdots\text{C5C6}$  (where  $\text{C5C6}$  is the midpoint of the  $\text{C5}-\text{C6}$  bond). Weak intermolecular  $\text{C}_{\text{phenyl}}-\text{H}\cdots O_{\text{hydroxyl}}$  interactions, with  $\text{H}\cdots\text{O}$  distances of 2.67 and 2.69 Å, connect neighboring chains in the *b* direction.

The ADDSYM option of program *PLATON* (Spek, 1998) revealed an approximate inversion center at position (3/8, 1/2, 0), indicating the existence of pseudosymmetry. The coordinates of molecule 2 are related to those of molecule 1 by the approximate relations:  $x_2 = 1/4 + x_1$ ,  $y_2 = y_1$  and  $z_2 = 1/2 - z_1$ . This corresponds to a non-crystallographic pseudo-glide-plane perpendicular to the *c* axis, with a translation of  $a/4$ . Molecule 2 is related to symmetry equivalents of molecule 1 by a pseudo-inversion center at (1/8, 0, 0) (which is a position equivalent to the pseudo-inversion center detected by *PLATON*), a pseudo-twofold screw axis along (*x*, 1/4, 1/4), with a translation vector of  $3a/4$ , and a pseudo-twofold screw axis along (3/8, *y*, 0), with a translation vector of  $b/2$ . These pseudosymmetry operations are local symmetry elements which relate molecule 1 to molecule 2.

The structure of (I) is closely related to the higher symmetric space group *Pnam* (non-standard setting of *Pnma*). If the *x* coordinates of the atoms of molecule 2 are shifted by  $-1/4$ , then the local pseudo-glide-plane in (I) is replaced by the mirror plane at  $z = 1/4$  of space group *Pnam*. A search of the Cambridge Structural Database (Allen, 2002) for crystal structures with space group *Pna2*<sub>1</sub>, reported with two independent molecules, revealed a number of other structures with pseudosymmetry relations. Examples are anthranilic acid (Brown & Ehrenberg, 1985), pyrazole (Larsen *et al.*, 1970; la Cour & Rasmussen, 1973), dimethylaminesulfur dioxide (Phillips *et al.*, 1996) and 3-amino-4-nitrofurazan (Batsanov & Struchkov, 1985). All these structures can be derived from the higher symmetric space group *Pnma* by translating some molecules with respect to their neighbors.

## Experimental

The methyl ester of the title compound was prepared following a procedure described by Smith *et al.* (1949) and Adlerová *et al.* (1964). 60 ml of an ethereal Grignard solution made from magnesium turnings (2.7 g, 111 mmol) and bromocyclopentane (16 ml, 150 mmol) was added dropwise to an ice-cooled solution of 2-oxo-2-phenylacetic acid methyl ester (7 g, 42.6 mmol) in 70 ml of dry diethyl ether. Aqueous work-up and flash chromatography (silica gel, petroleum ether/ethyl acetate, 20:1) afforded 3.3 g (33%) of 2-cyclopentyl-2-hydroxy-2-phenylacetic acid methyl ester. The title compound, (I), was obtained by hydrolysis of this ester with lithium hydroxide monohydrate in a mixture of THF and water (1:1). Single crystals of (I) were recrystallized from diethyl ether.

### Crystal data

$\text{C}_{13}\text{H}_{16}\text{O}_3$	Mo $K\alpha$ radiation
$M_r = 220.26$	Cell parameters from 194 reflections
Orthorhombic, <i>Pna2</i> <sub>1</sub>	$\theta = 3-23^\circ$
$a = 11.1981(14)$ Å	$\mu = 0.09$ mm <sup>-1</sup>
$b = 8.4864(15)$ Å	$T = 142(2)$ K
$c = 23.853(3)$ Å	Rod, colorless
$V = 2266.8(6)$ Å <sup>3</sup>	$0.50 \times 0.45 \times 0.40$ mm
$Z = 8$	
$D_x = 1.291$ Mg m <sup>-3</sup>	

### Data collection

Siemens SMART 1K CCD diffractometer	3878 independent reflections
$\omega$ scans	3597 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan ( <i>SADABS</i> , Sheldrick, 2000)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.935$ , $T_{\text{max}} = 0.964$	$\theta_{\text{max}} = 32.0^\circ$
37 645 measured reflections	$h = -16 \rightarrow 16$
	$k = -11 \rightarrow 10$
	$l = -34 \rightarrow 35$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.35P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.39$ e Å <sup>-3</sup>
3878 reflections	$\Delta\rho_{\text{min}} = -0.23$ e Å <sup>-3</sup>
294 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0043 (10)

**Table 1**

Hydrogen-bonding geometry (Å, °).

 $Cg_2$  is the centroid of the phenyl ring of molecule 2 and C5C6 is the midpoint of the C5–C6 bond.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H01 $\cdots$ O5 <sup>i</sup>	0.84	2.00	2.794 (2)	156
O3–H03 $\cdots$ O1 <sup>ii</sup>	0.84	1.83	2.664 (2)	173
O4–H04 $\cdots$ O2	0.84	2.00	2.762 (2)	151
O6–H06 $\cdots$ O4 <sup>ii</sup>	0.84	1.78	2.621 (2)	174
C5–H5 $\cdots$ Cg <sub>2</sub> <sup>iii</sup>	0.95	2.82	3.521 (2)	132
C18–H18 $\cdots$ C5C6	0.95	2.73	3.582 (2)	150
C6–H6 $\cdots$ O1 <sup>iv</sup>	0.95	2.67	3.587 (2)	163
C19–H19 $\cdots$ O4 <sup>iv</sup>	0.95	2.69	3.601 (2)	160

Symmetry codes: (i)  $x - \frac{1}{2}, \frac{3}{2} - y, z$ ; (ii)  $\frac{1}{2} + x, \frac{3}{2} - y, z$ ; (iii)  $x - \frac{1}{2}, \frac{1}{2} - y, z$ ; (iv)  $x, y - 1, z$ .

Equivalent reflections, including Friedel opposites, were merged, since anomalous dispersion effects are negligible. The systematic absences showed the space group to be either  $Pna2_1$  or  $Pnam$  (non-standard setting of  $Pnma$ ). The almost centrosymmetric distribution of the  $E$  values and the value of  $Z$  ( $Z = 8$ ) are indications of the latter space group. All attempts to determine the structure in space group  $Pnam$ , however, failed. Consequently, the structure was determined in the non-centrosymmetric space group  $Pna2_1$ . The resulting structure can not be transformed to the centrosymmetric space group  $Pnam$ . The H atoms were located in a difference Fourier synthesis. They were refined with fixed individual displacement parameters [ $U_{iso}(H) = 1.2U_{eq}(C)$ ,  $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$  and  $U_{iso}(H) = 1.5U_{eq}(O)$ ] using a riding model, with fixed distances: H–O = 0.84 Å, H–C(phenyl) = 0.95 Å, H–C(secondary) = 0.99 Å and H–

C(primary) = 1.00 Å. The hydroxyl and carboxyl H atoms were allowed to rotate about the O–C bonds.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

## References

- Adlerová, E., Vejdělková, P. & Protiva, M. (1964). *Collect. Czech. Chem. Commun.* **29**, 97–119.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Batsanov, A. S. & Struchkov, Y. T. (1985). *Zh. Strukt. Khim.* **26**, 65–69.
- Brown, C. J. & Ehrenberg, M. (1985). *Acta Cryst.* **C41**, 441–443.
- Cour T. la & Rasmussen, S. E. (1973). *Acta Chem. Scand.* **27**, 1845–1854.
- Guy, J. J. & Hamor, T. A. (1973). *J. Chem. Soc. Perkin Trans. 2*, pp. 1875–1879.
- Larsen, F. K., Lehmann, M. S., Sjøtofte, I. & Rasmussen, S. E. (1970). *Acta Chem. Scand.* **24**, 3248–3258.
- Phillips, J. A., Britton, D. & Leopold, K. R. (1996). *J. Chem. Crystallogr.* **26**, 533–538.
- Sheldrick, G. M. (1996). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2000). *SADABS*. University of Göttingen, Germany.
- Siemens (1995). *SMART* (Version 4.05) and *SAINT* (Version 4.05). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Smith, H. A., Alderman, D. M. Jr, Shacklett, C. D. & Welch, C. M. (1949). *J. Am. Chem. Soc.* **71**, 3772–3776.
- Spek, A. L. (1998). *PLATON*. Utrecht University, The Netherlands.